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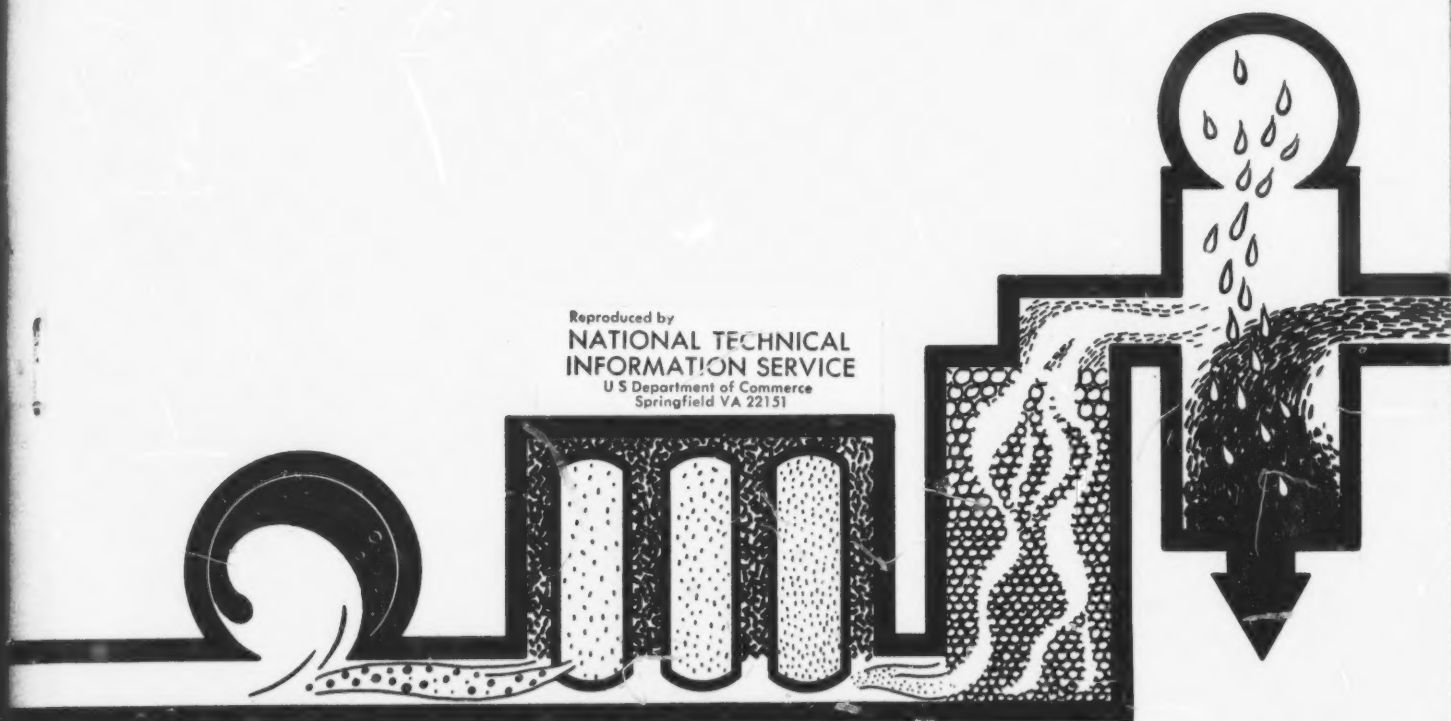


WATER POLLUTION CONTROL RESEARCH SERIES ● ORD-17010EER06/70

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**MOBILE PILOT PLANT
FOR
REMOVAL OF PHOSPHATE FROM WASTEWATERS
BY ADSORPTION ON ALUMINA**

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16. Abstract: Alumina, a phosphorus-selective adsorbent, was used in a tertiary chemical process for phosphorus removal. A mobile pilot plant designed and built on the basis of laboratory results was fitted into a standard forty-foot van trailer. The secondary effluent was passed through a thirty inch multimedia filter bed and stored in an exterior 2500 gallon surge tank. Then the filtered sewage was pumped through one or more of three alumina beds approximately forty inches in depth and three feet in diameter. Phosphorus loading of the alumina columns can be in a downflow or upflow direction with the columns in series or parallel. Phosphorus-free effluent was stored in another tank for use as alumina column wash solution. 800 gallons of 1M NaOH was recirculated to remove absorbed phosphorus in an alumina column taken off the line. Valving and column washing was pH controlled. $\text{Ca}(\text{OH})_2$ was added to the NaOH solution in a mixing tank to precipitate phosphorus in the form of hydroxyapatite. The solids were removed in a continuous centrifuge and the Na(OH) returned for reuse. Initial operating costs were approximately 5.8 cents/1000 gallons. (Smith-Texas)

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RESEARCH REPORT
RESEARCH TO DEVELOP AND DEMONSTRATE A
MOBILE PILOT PLANT FOR REMOVAL OF SOLUBLE
PHOSPHORUS FROM WASTEWATERS BY ABSORPTION ON
ALUMINA COLUMNS

By

L. L. Ames, Jr.

To

The Advanced Waste Treatment Research Laboratory

Robert A. Taft Water Research Center

This report is submitted in
fulfillment of Contract No.
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Northwest.

U. S. Department of the Interior
Federal Water Pollution Control Administration
Cincinnati, Ohio

June 1970

A B S T R A C T

Alumina, a phosphorus-selective adsorbent, was used in a tertiary chemical process for phosphorus removal. A mobile pilot plant was designed and built based on laboratory results. The pilot plant was designed to fit into a standard, used, forty-foot van trailer.

The secondary effluent was passed through a thirty inch multimedia filter bed and stored in an exterior 2500 gallon surge tank in the form of a collapsible plastic swimming pool. From this tank, the filtered sewage was pumped through one or more of three alumina beds approximately forty inches in depth and three feet in diameter. The phosphorus loading of the alumina columns can be in a downflow or upflow direction with the columns operated in series or parallel configurations. Phosphorus-free column effluent was stored in another collapsible pool for use as alumina column wash solution. After phosphorus loading, an alumina column was taken off the line and 800 gallons of 1M NaOH used to remove the absorbed phosphorus by recirculation. Valving was controlled by column effluent pH measurements. Column washing following the elution cycle, likewise, was pH controlled. After a water wash, the alumina column can be put back on the line. The used NaOH solution was pumped from the caustic makeup tank to the mixing tank, and $\text{Ca}(\text{OH})_2$ added with stirring to precipitate phosphorus in the form of solid hydroxyapatite. The solids were removed in a continuous centrifuge and the NaOH returned to the makeup tank for reuse. Initial operating costs were approximately 5.8 cents/1000 gal; nearly the same as the operating cost for a 10 MGD plant estimated from laboratory results.⁽¹⁾

This report was submitted in fulfillment of Contract Number 14-12-566, Program No. 17010 EER, between the Federal Water Quality Administration and Battelle-Northwest.

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SECTION I

CONCLUSIONS AND RECOMMENDATIONS

The alumina pilot plant, as designed and constructed, efficiently removes phosphorus from secondary sewage effluents.

Based on initial pilot plant data, the chemical and filtration costs for the alumina process has not changed appreciably from the 3 to 6 cents/1000 gal of the laboratory study. The economics are competitive, especially on trickling filter plant operations that do not readily allow chemical additions during process operations.

Additional operating data should be obtained to verify initial results. It is recommended that the present trained crew operate the alumina pilot plant in several geographical locations to determine the effects of influent composition and other operating conditions on process performance and economics, and to demonstrate the process to interested personnel.

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2. ESTIMATED MATERIALS OPERATING COSTS ASSUMING 98% P
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SECTION I

CONCLUSIONS AND RECOMMENDATIONS

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SECTION II

INTRODUCTION

The laboratory scale phase of a study of phosphorus adsorption on alumina was recently completed with encouraging results⁽¹⁾. The removal of phosphorus from secondary sewage effluent was successfully demonstrated using one liter alumina columns. Approximately 400 column volumes of Richland, Washington secondary effluent were required to reach 10% phosphorus breakthrough. If two columns were operated in series, more than 600 column volumes could be loaded on the first column before phosphorus elution would be required. The alumina columns were satisfactorily regenerated with NaOH in a half to one hour. An acidic wash was not required to recondition the column. Alumina dissolution in a 1M NaOH solution averaged 0.33 wt. %/hr, or about 300 elution cycles of one hour duration would be required to completely dissolve the alumina in the column as sodium aluminate. The above value may have been unrealistically high because the bulk of the alumina lost can be attributed to an initial physical loss of alumina fines, rather than averaging the loss over a period of time as was done in the study.

Sodium hydroxide physical losses are about 5% of a column volume. Other losses include that hydroxyl ion necessary to displace PO_4^{-3} , HCO_3^- and other anions adsorbed on the column. Calcium hydroxide can be added to the used regenerant NaOH to precipitate solid calcium hydroxylapatite, thus recovering the NaOH for reuse. Much of the coloring matter was removed with the solids.

Treatment costs were estimated from the above laboratory data as between five and six cents/1000 gal for a ten million gal/day plant.⁽¹⁾

At the conclusion of the above laboratory study, it became apparent that several operating parameters should be measured on a larger scale for realistic cost results. For this reason, the phosphorus removal process was recommended for scaleup to a portable pilot plant size with shakedown at Richland. This procedure also would allow acquisition of operating data at several field sites with different sewage influent compositions.

The design, construction, shakedown and three weeks of operation of the pilot plant at the Richland Municipal trickling filter plant are described in this report.

SECTION III
PILOT PLANT DESIGN

A mobile pilot plant was designed and built for larger scale phosphorus removal studies based on the laboratory results. The pilot plant was designed to fit into a standard forty foot van trailer as shown in Figure 1. The secondary effluent is passed through a thirty inch multimedia filter bed and stored in an exterior 2500 gallon surge tank in the form of a collapsible plastic swimming pool. From this tank, the filtered sewage is pumped through one or more of three alumina columns containing 150 to 160 gallons of 20 to 50 mesh, high surface area, alumina. The alumina beds are of fiberglass approximately 35 inches in diameter, as is the filterbed column. After passing through the alumina bed, some of the effluent is retained in a phosphate-free water storage tank for column backwashing. Storage of the caustic regenerant solution is in the approximately 800 gallon makeup tank at the rear of the van trailer. From this tank, the caustic regenerant is pumped through a phosphorus loaded alumina column, and the used solution sent to the mixer and settler tank located in the forward section of the trailer. Calcium hydroxide is added and the mixture stirred until the phosphorus content is less than 30 mg P/l. The centrifuge is used to remove the hydroxylapatite solid from the liquid. The regenerant caustic then passes into a small surge tank under the centrifuge, and is pumped back to the makeup tank for storage and reuse. A small analytical laboratory facility is located in the front of the van to allow rapid phosphorus analyses in support of the loading and regeneration activities. The pumps are located in a rack in the center of the van.

Structural modifications to the van trailer itself included fabrication of two hatches, one over the alumina columns and one over the filter column. The hatches are necessary to allow access to the columns if repair or maintenance work is required. Two doors in the rear of the trailer were designed to give access to plumbing behind the alumina columns. The door at the front of the van allows access to the rest of the pilot plant. Metal ladders were fabricated for the front and a rear doorway. A ladder is permanently attached to the trailer for access to the roof.

The isometric details of the plumbing system are given in Figure 2. Nearly all of the piping is polyvinyl chloride plastic with some stainless steel fittings. Most surfaces normally in contact with caustic solution are stainless steel, fiberglass construction, plastic, or coated with caustic-resistant primer and paint.

The plumbing design was made quite flexible as to mode of operation by the generous use of quick-disconnect hose fittings, while at the same time, avoiding undue piping bulk by use of multiport valves. The multiport valves are mild steel, but no problems are expected with caustic. The attack of dilute caustic (about 4%) on mild steel is relatively slow and can be tolerated in this instance.

The design and construction phases of the pilot plant lasted approximately nine and one half months. Several photographs of the finished pilot plant are presented in the following figures. An exterior view of the pilot plant with the portable tanks (swimming pools) set up is given in Figure 3. Figure 4 shows an interior view of the pilot plant looking toward the rear. The multimedia filter column is on the

left with multiport valves and alumina columns in the background. An interior view toward the front of the pilot plant is given in Figure 5. The pump rack, filter column and lab bench are visible. Another interior view of the pilot plant looking toward the rear is given in Figure 6. The centrifuge, mixing tank and filter column can be seen.

The alumina used in the pilot plant columns was a proprietary product of the Aluminum Company of America. The surface area of the alumina was greater than $200 \text{ m}^2/\text{g}$, and was furnished in a 20 to 50 mesh size range. All of the alumina columns had a cross-sectional area of 6.67 ft^2 . The alumina volumes are given in Figures 7 through 12. Alumina bed depths varied from 38 to 42 inches after an initial backwash to remove any fines (<60 mesh) generated during column filling.

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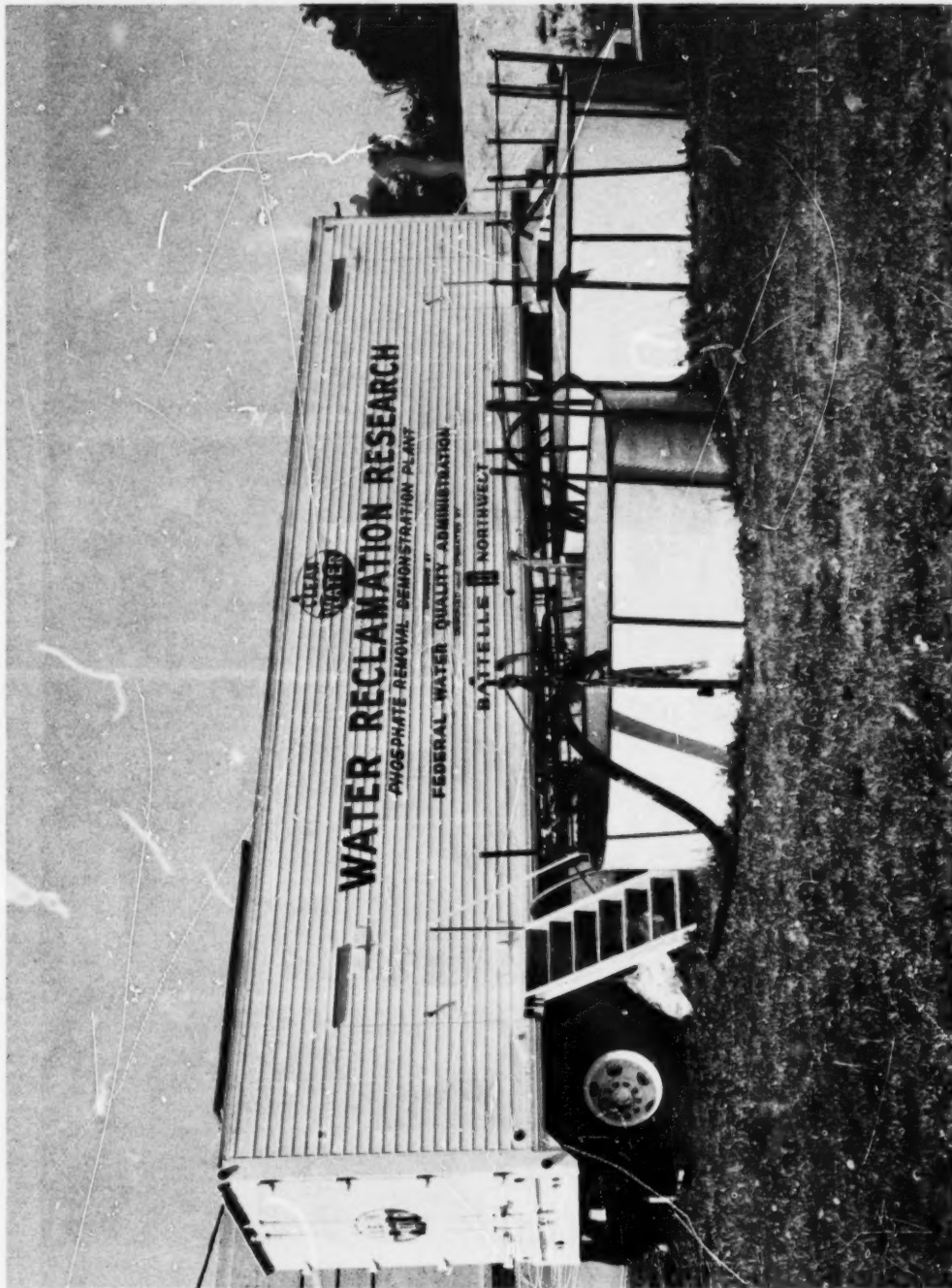


FIGURE 3. An exterior view of the alumina pilot plant.

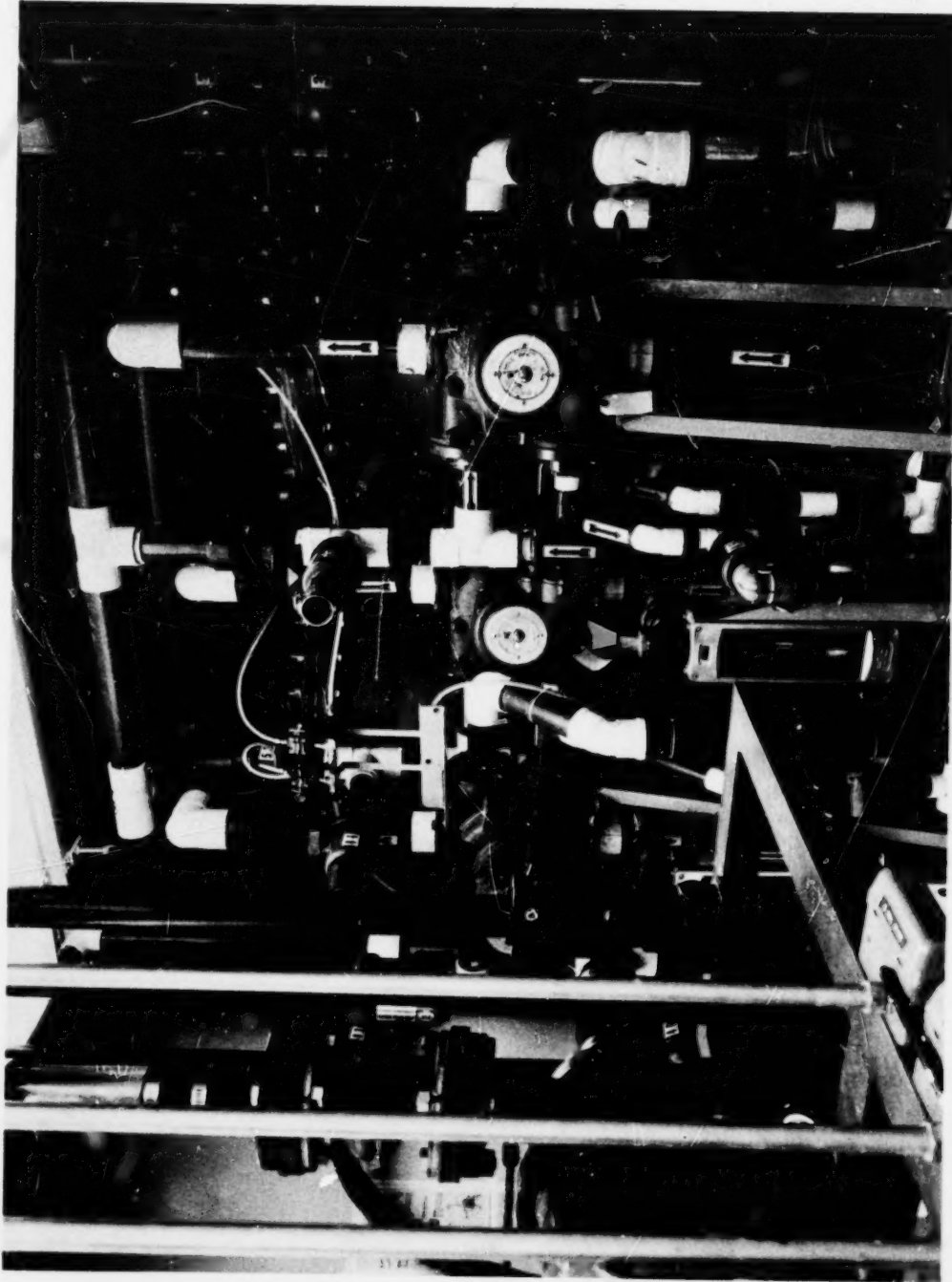


FIGURE 4. An interior view of the pilot plant looking toward the rear from just in front of the filter column.

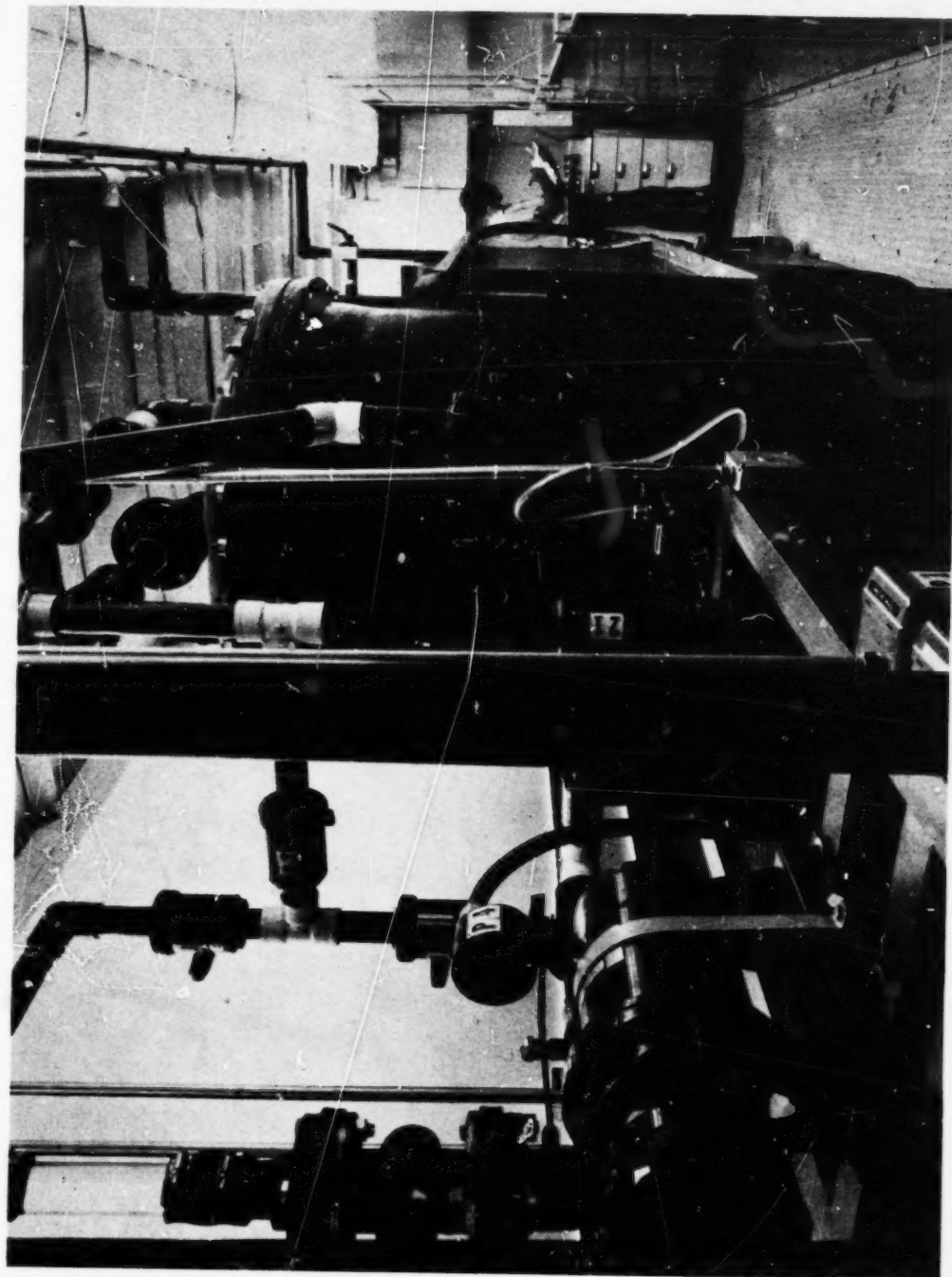


FIGURE 5. An interior view of the pilot plant looking toward the front from along side the pump rack.

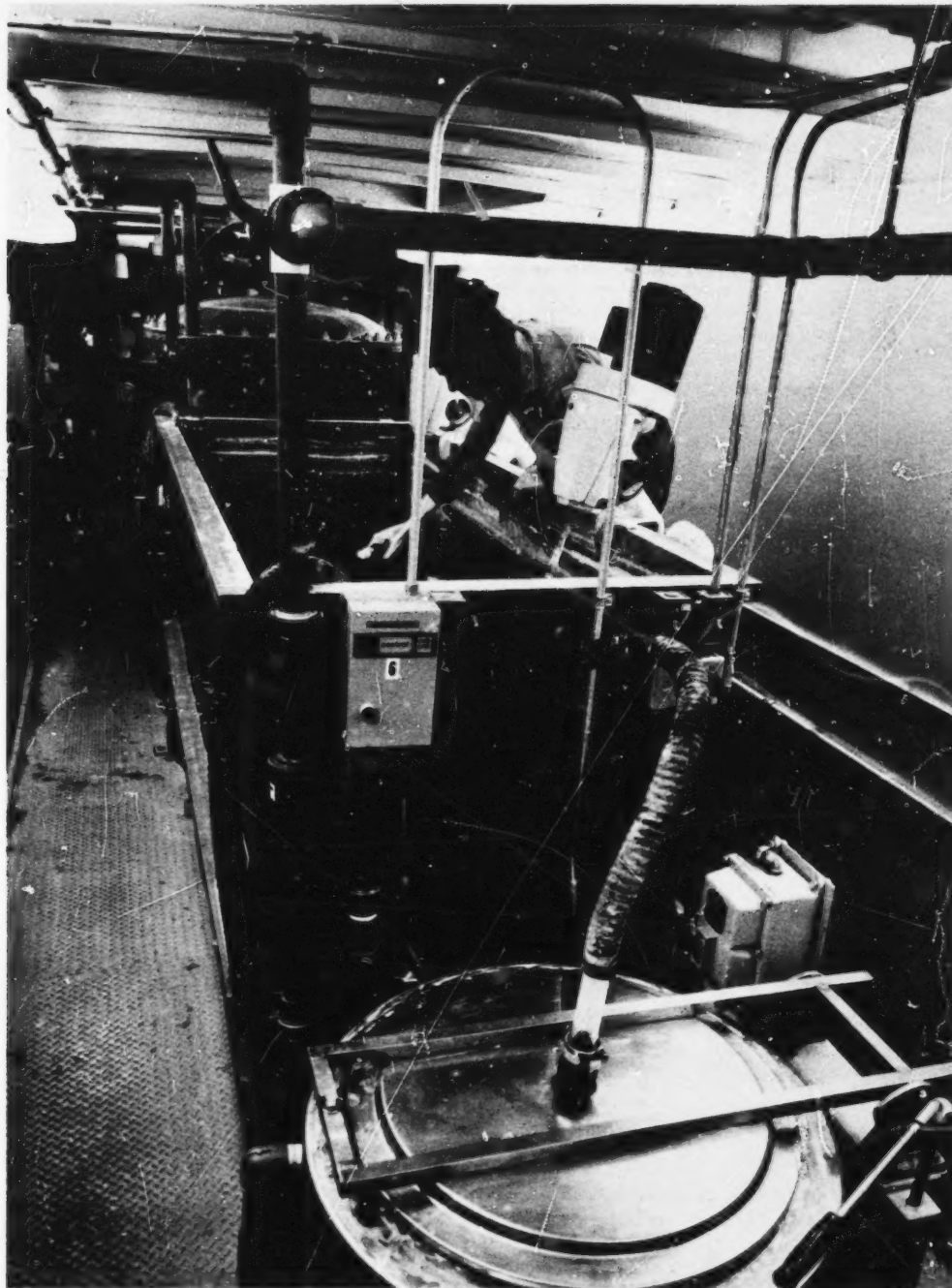


FIGURE 6. An interior view of the pilot plant from the front door looking toward the back of the trailer across the centrifuge and mixing tank.

SECTION IV

PILOT PLANT OPERATION

Upon completion of the pilot plant at West Richland, it was moved to the Richland municipal sewage treatment plant, a trickling filter operation, for shakedown. The remainder of April and part of May was utilized in setting up the portable tanks, devising operating procedures, training an operating crew, and locating and repairing the shortcomings of the as-built mechanical and plumbing systems. Approximately three weeks were spent in continuous operation with the last two weeks of the contract spent in repainting, accomplishing repairs, installation of an automatic flow control and recording system, and generally preparing the pilot plant for moving or storage. This section of the report will be chiefly concerned with the period of continuous pilot plant operation at the Richland Municipal trickling filter plant.

The compositional range of the Richland secondary effluent is given in Table 1. The compositional range represents the alumina column influent or feed. Greater than 95% of the total phosphorus in the secondary effluent above was in the form of orthophosphate (PO_4^{-3}). The improved Edwards phosphorus determination method⁽²⁾ was used for phosphorus analysis. This method has the advantage of a stable color over several hours, allowing for more flexible trailer operations.

During the three week period of continuous operation, the pilot plant was operated on a five-day, twenty-four hour schedule. The alumina columns had been individually loaded with phosphorus and eluted several times during training operations.

TABLE 1

RICHLAND SECONDARY EFFLUENT COMPOSITION

<u>Constituent</u>	<u>mg/l Range</u>
Na ⁺	40-60
K ⁺	12-15
NH ₄ ⁺	10-14
Mg ⁺⁺	6-9
Ca ⁺⁺	25-40
NO ₃ ⁻	11
P	8-12
Alkalinity as CaCO ₃ equivalent	180-190
pH	7.2-7.8

Loading Operations

Two columns were operated in series during this period, while the third column was being regenerated. The first in the series of loading operations is shown in Figure 7 in terms of column volumes. The effluent from column B comprised the influent for column C, both in downflow operation. Phosphorus leakage was between one and two percent. About 550 column volumes to 10% phosphorus breakthrough were obtained on column B. Fifty percent breakthrough was reached at 800 column volumes for column B. C column effluent remained below two percent phosphorus breakthrough to 400 column volumes. An incomplete backwash of column C caused the initially high phosphorus values. Column B was taken off the series at 854 column volumes of throughput.

The use of column volumes is legitimate for the first two columns because both columns were recently regenerated before being placed on line. Normally, the first column, column B in this case, is partially loaded with phosphorus from being used as the second column in series on the previous series run. Consequently, these data, and all data following, are also given in gallons of throughput rather than in terms of column volumes.

The data of Figure 7 are presented in Figure 8 in terms of gallons of throughput, rather than column volumes, so that they are consistent with the remainder of the series operation data. Consecutive phosphorus loadings in series operation are shown in Figures 9, 10, 11 and 12. It is difficult to assess these data in terms of column volumes because column C of Figure 8, for example, was moved from the second column in line, receiving a relatively low phosphorus influent, to the first column

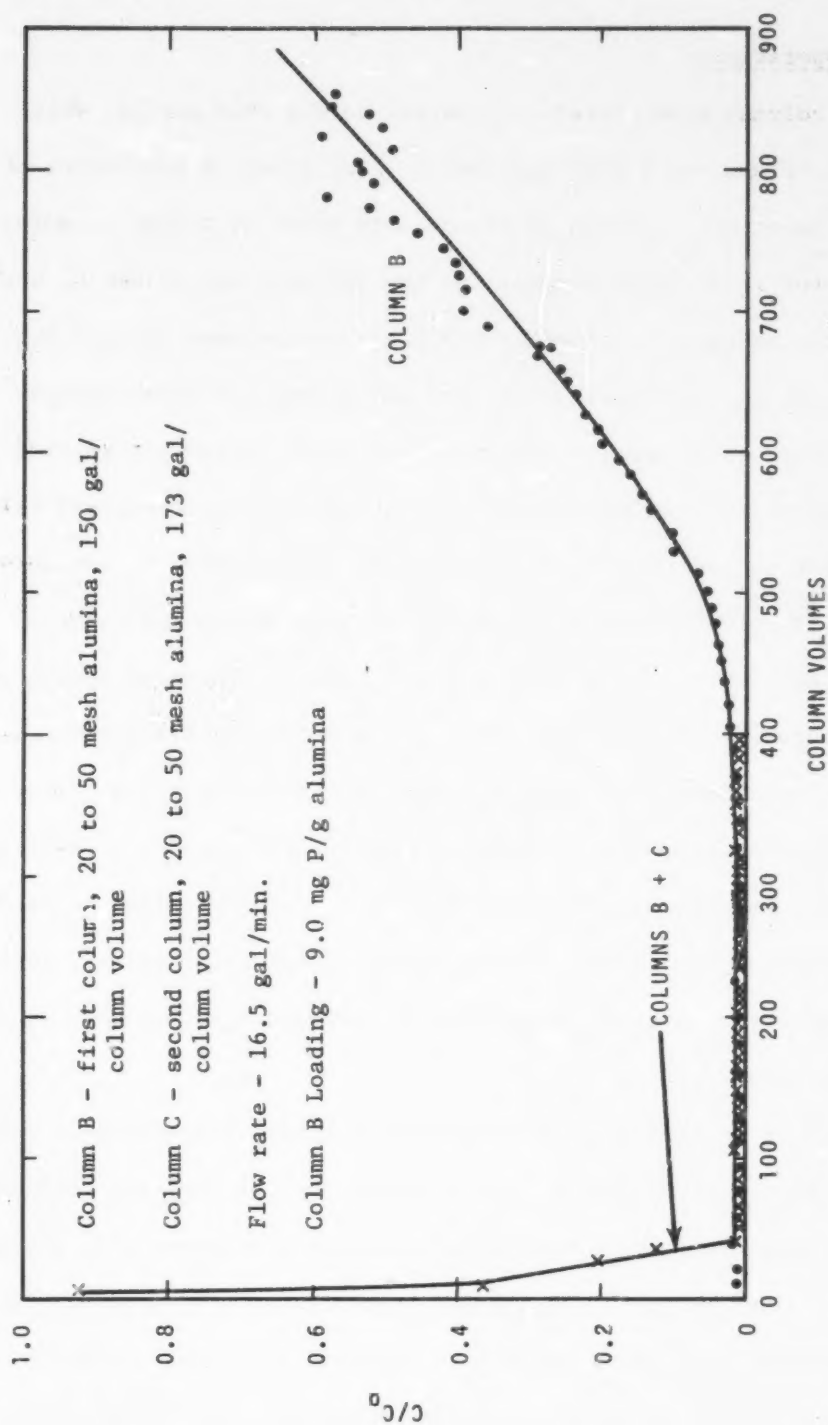


FIGURE 7. Series, downflow loading operation of columns B and C. Throughput is plotted as column volumes, C/C_0 is the fraction of influent phosphorus concentration found in the column effluent.

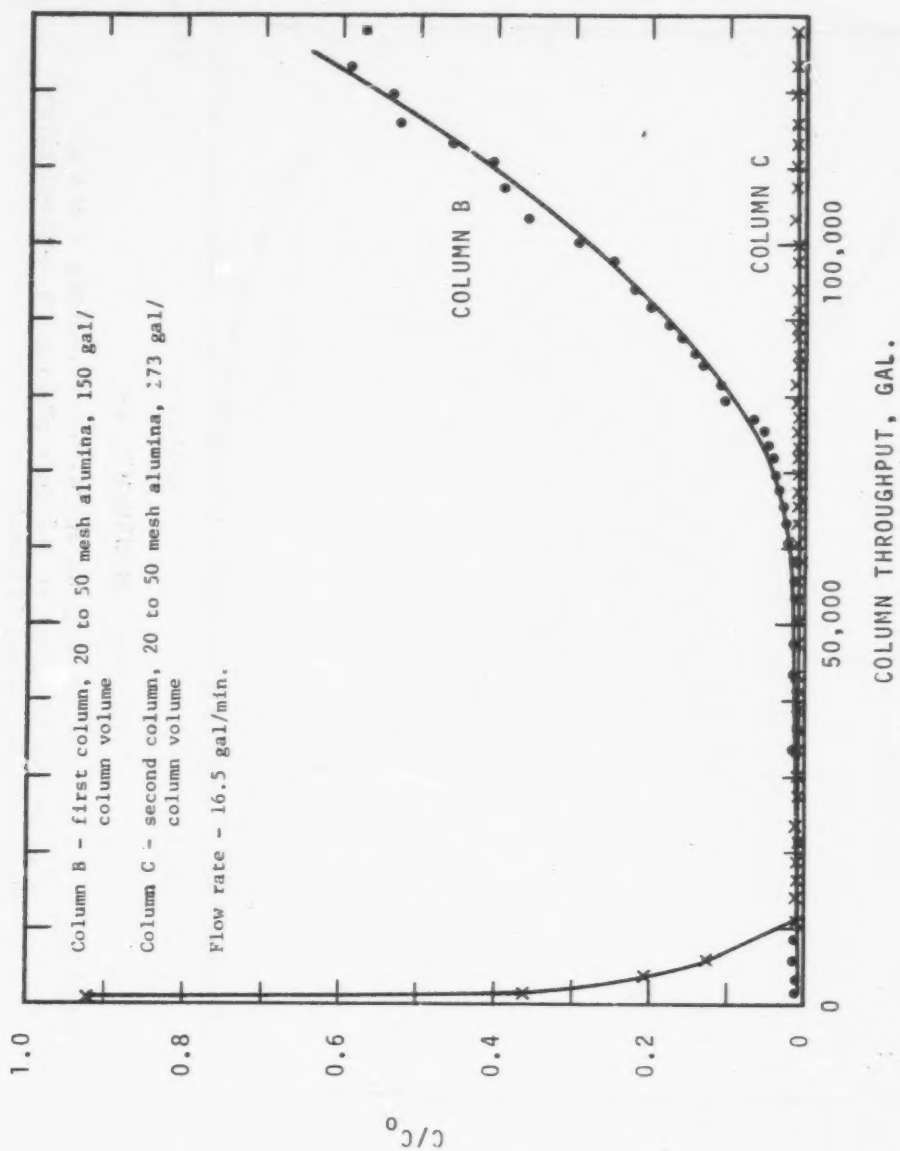


FIGURE 8. Series, downflow loading operation of columns B and C. Data is the same as for Figure 7 except throughput is in gallons. C/C_0 is the fraction of influent phosphorus concentration found in the column effluent.

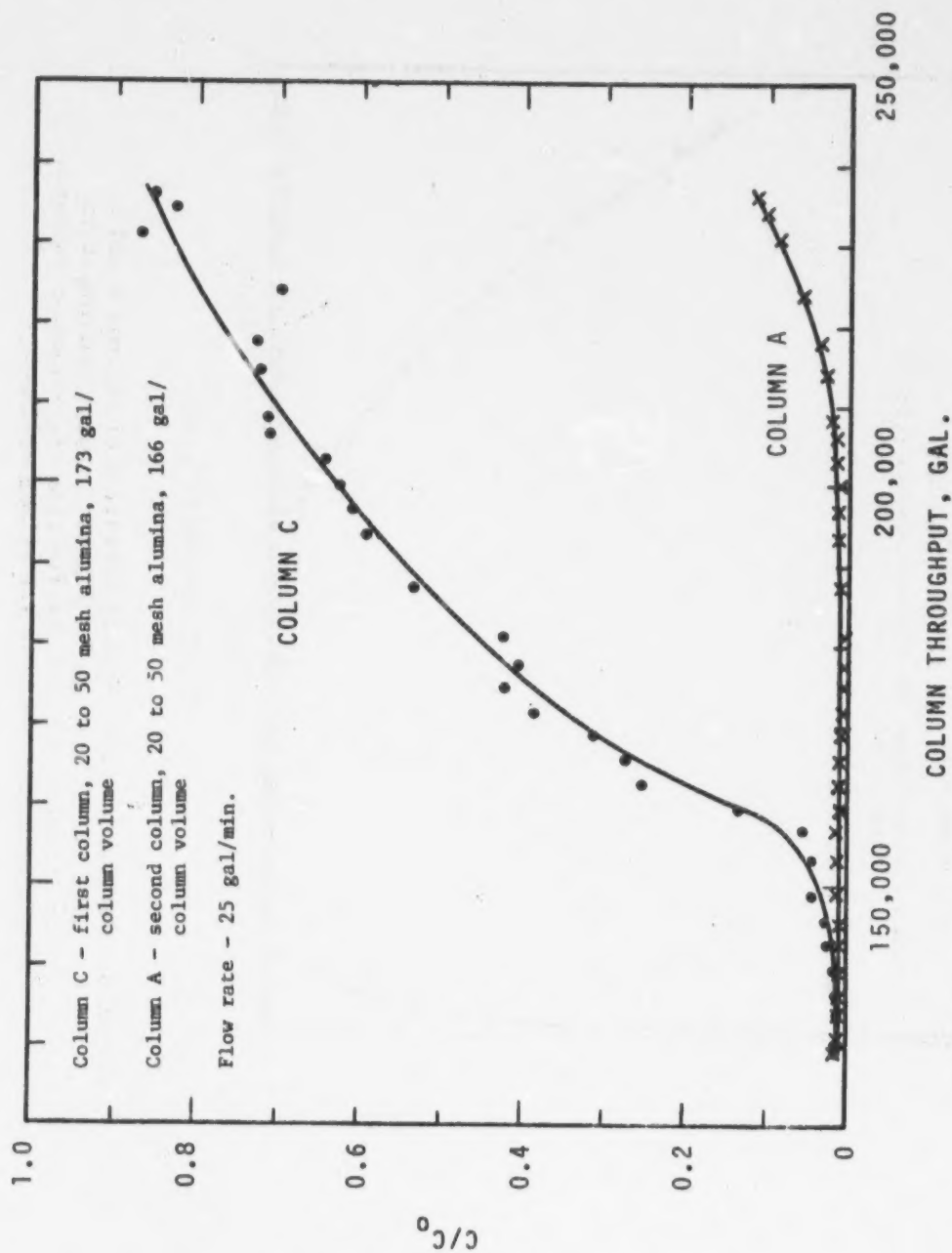


FIGURE 9. Series, downflow loading operation of columns C and A.
 C/C_0 is the fraction of influent phosphorus concentration
found in the column effluent.

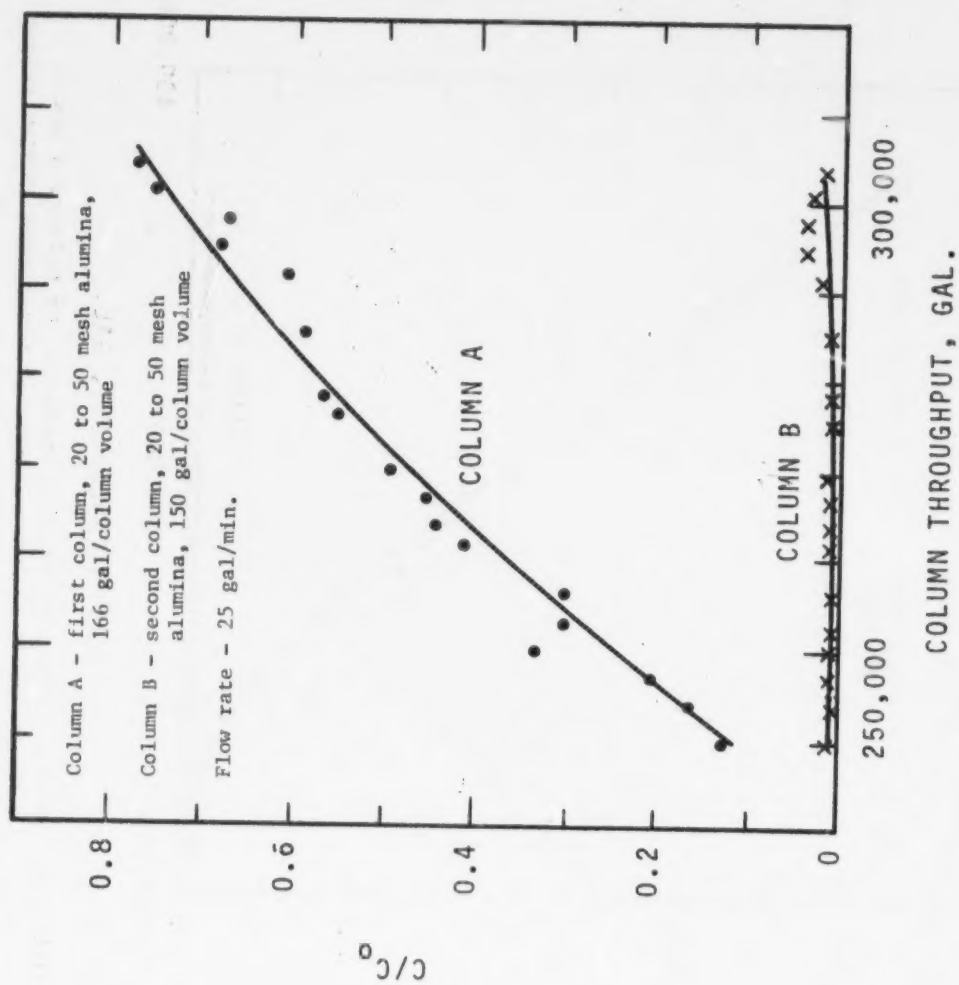


FIGURE 10. Series, downflow loading operation of columns A and B.
C/C₀ is the fraction of influent phosphorus concentration found in the column effluent.

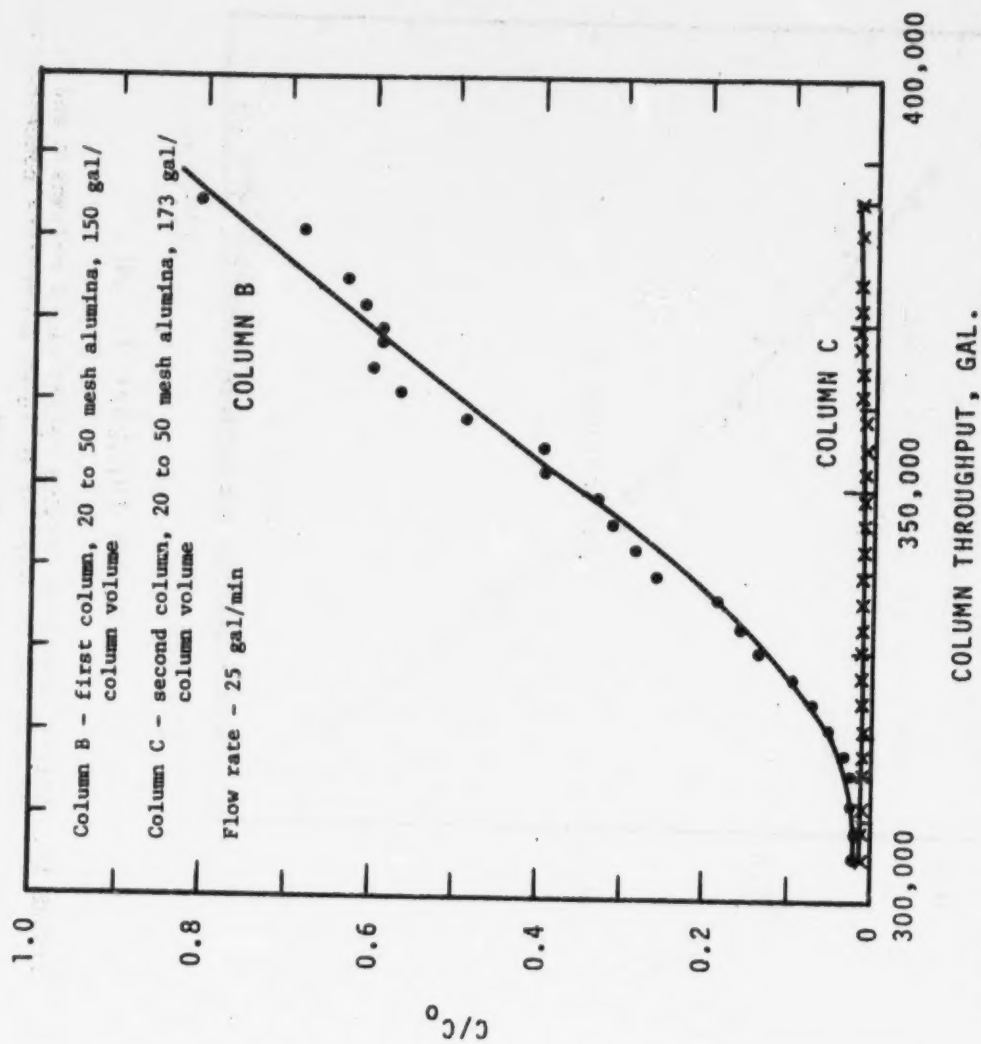


FIGURE 11. Series, downflow loading operation of columns B and C.
 C/C_0 is the fraction of influent phosphorus concentration
found in the column effluent.

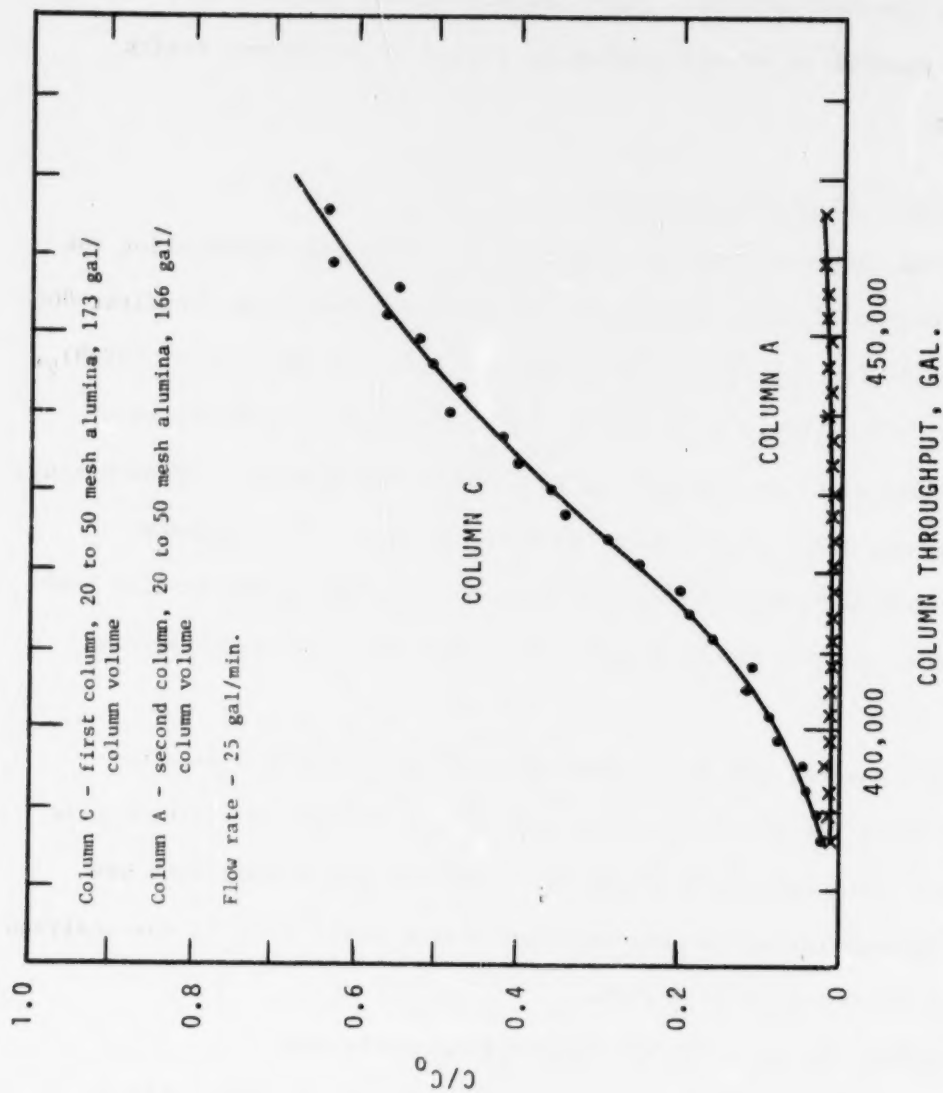


FIGURE 12. Series, downflow loading operation of columns C and A. C/C_0 is the fraction of influent phosphorus concentration found in the column effluent. Not enough process data was obtained to evaluate the effects of flow rate on product quality.

in line in Figure 9, receiving all the phosphorus in the influent. The column C apparent throughput was 1422 column volumes. However, presenting the data in this manner does allow for easier assessment of costs per thousand gallons. Approximately 465,000 gallons were treated with 98% phosphorus removal during the period of continuous series operation.

Regeneration and Ca(OH)_2 Addition

During continuous series operation, a method for regenerating the phosphorus-loaded alumina was used that involved retaining the first 800 gal of washwater following caustic regeneration and addition of Ca(OH)_2 , and using this average 0.1M NaOH to initially pass through the next loaded alumina column, discarding it ahead of the regular 1.0M NaOH single pass regeneration. This process results in cost savings because considerable phosphorus is removed from the alumina by the initial backwash water, and the column also is partially saturated with hydroxyl ions as well.

Eight hundred gallons of approximately 1.0M NaOH was used to finally strip the phosphorus from the alumina columns in a single pass operation. The caustic solution in the makeup and storage tank was pumped through the column and into the mixing tank. Here it was analyzed for phosphorus and Ca(OH)_2 added.

Briefly, the steps in the regeneration cycle are:

1. backwashed with 200 to 400 gal of phosphorus-free water at 10 gal/min to remove any solids on top of alumina column;
2. after loading, the alumina column was backwashed at 10 gal/min with 800 gallons of previously retained, 0.05 to 0.1M NaOH, 5 to 300 mg P/l water which was recirculated to input;

3. regeneration was downflow at 25 gal/min with 800 gal of 1.0M NaOH from the makeup and storage tank;
4. the used regenerating solution was sent to the mixing tank, the appropriate amount of Ca(OH)_2 added, makeup NaOH was added to bring the solution to 1.0M NaOH, the solution settled and was centrifuged to remove solids and pumped back to the storage tank;
5. the newly regenerated alumina column was then backwashed at 25 gal/min with 800 gal of water which was stored in the empty mixing tank to await step (1) in the next regeneration cycle, followed by 3000 gal of phosphorus-free washwater recirculated to process input; and,
6. the alumina column was returned as needed to downflow operation on the loading cycle.

A graph was made up to estimate the amount of Ca(OH)_2 to add to each 800 gallon batch of used eluting solution, and the graph is given in Figure 13. All operational analytical data is obtained as mg $\text{PO}_4/1$ and from this, the content of the solution in pounds of $\text{PO}_4/800$ gal and pounds of Ca(OH)_2 required to precipitate this as $\text{Ca}_5(\text{PO}_4)_3\text{OH}$ can be read. This amount of Ca(OH)_2 represents a minimum to be added because carbonate present in the solution and added during mixing is ignored, as well as other phosphate compounds. Figure 14, for example, shows PO_4 removal from B column regenerant with 20 lbs of added Ca(OH)_2 . The regenerant contained 626 mg P/1. Figure 13 shows 12.8 lbs $\text{PO}_4/800$ gal requiring a minimum of 18.2 lbs of Ca(OH)_2 . Since the commercial lime is not much more than 90% Ca(OH)_2 , just 20 lbs would be required. As can be seen from Figure 14, 20 lbs was not quite enough Ca(OH)_2 . On the other hand, when 30 lbs

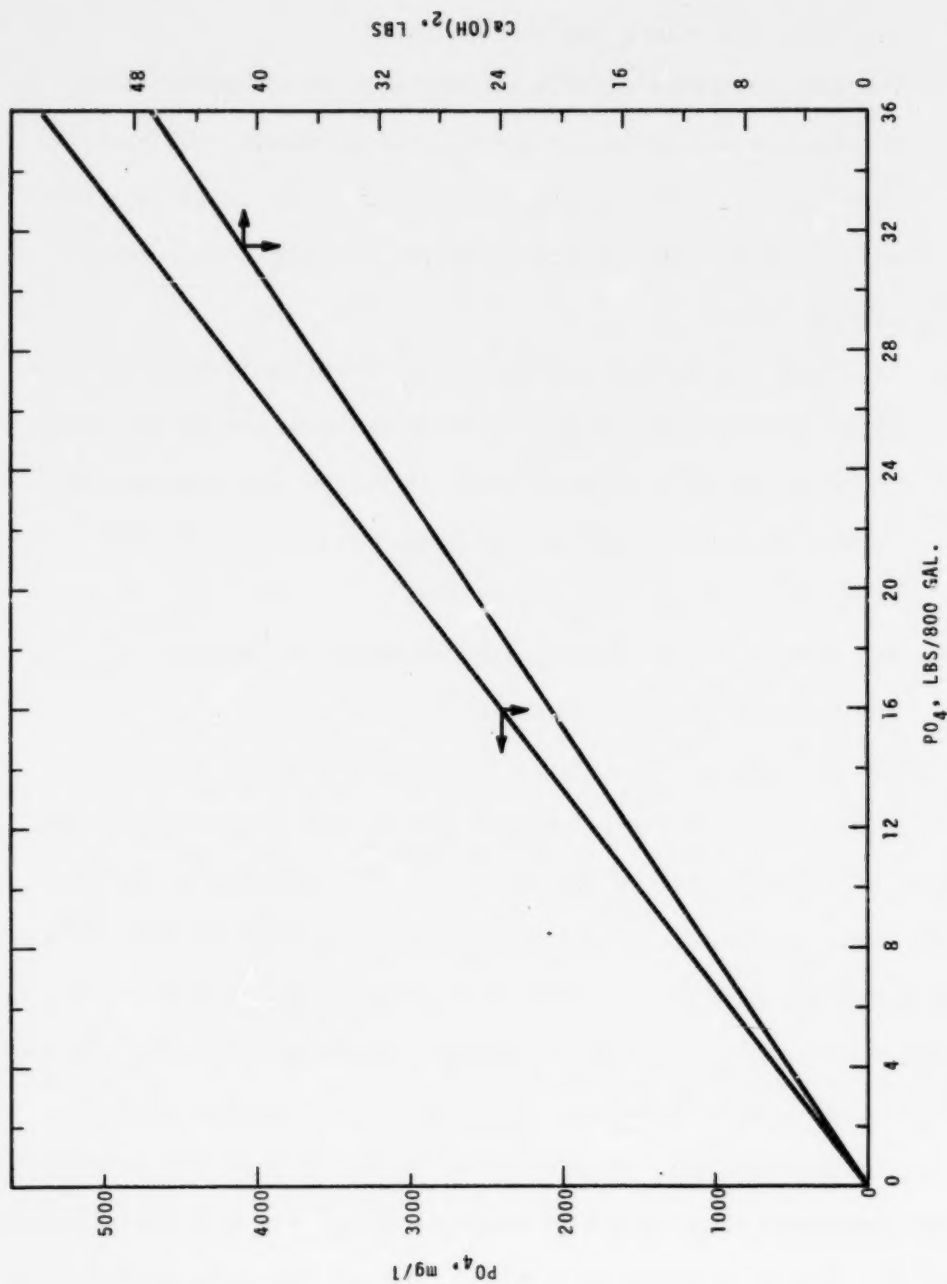


FIGURE 13. A working graph of the relationship between phosphate in the used eluting solution in mg/l, phosphate in lbs/800 gal of eluting solution, and required Ca(OH)_2 in lbs assuming $\text{Ca}_5(\text{PO}_4)_3\text{OH}$ as the solid product.

of lime were added to a used regenerant containing 496 mg P/l (10.1 lbs PO_4 /800 gal), requiring 15.2 lbs $\text{Ca}(\text{OH})_2$. PO_4 removal was good and continued at a good rate until the mixing was terminated, as seen in Figure 15. Excess $\text{Ca}(\text{OH})_2$ was identified by X-ray diffraction in the centrifuged solids, along with CaCO_3 and hydroxylapatite. The use of CaO did not materially improve the reaction rate. Even longer reaction times would be required if the $\text{Ca}(\text{OH})_2$ regenerant were not both stirred and recirculated through the centrifuge pump and back into the mixing tank.

The reacted $\text{Ca}(\text{OH})_2$ products (apatite-calcite) settled readily to the bottom of the mixing tank in a half hour or less after the mixers were stopped. The coffee-colored, used caustic solution, after settling of the apatite-calcite mixture, can be viewed in Figure 6. The settled caustic was being sampled before centrifugation commenced. The solution is a cloudy white when the mixers are in operation.

A total of 140 pounds of $\text{Ca}(\text{OH})_2$ were added to the caustic regenerant solutions following the column loading operations shown in Figures 8, 9, 10, 11 and 12, or an average of 28 pounds of $\text{Ca}(\text{OH})_2$ per column regeneration. From the 140 pounds of added $\text{Ca}(\text{OH})_2$, approximately 189 pounds of solids were generated, chiefly hydroxylapatite, calcite and a small amount of excess $\text{Ca}(\text{OH})_2$.

Liquid wastes generated by the alumina process for each cycle included 400 gallons of low phosphorus backwash water following column loading, 800 gallons of high phosphorus, relatively high caustic (0.05N to 0.1N) backwash preceeding elution with 1.0N NaOH , and another 3000 gallons of high-to-low phosphorus backwash water following the elution. The first 400 gallons of low phosphorus backwash water (3 to 8 mg P/l) can be discarded with the column effluent without materially

effecting phosphorus decontamination, since it constitutes less than half a percent of the total volume treated (less than 2000 gal for the five regeneration cycles). The backwashes of step (2), however, contain from 300 mg P/l initially to 5 mg P/l; and, therefore, will have to be at least partially recycled to the head end of the plant. For the five regeneration cycles, the two high phosphorus backwashes constitute four percent of the total volume of secondary sewage treated, or about 19,000 gallons of the 465,000 gallons treated. The normal buffering capacity of the sewage will make treatment of this waste for OH^- removal unnecessary.

Initial Chemical Cost Estimate

The makeup requirements for caustic amounted to approximately 25 lbs/800 gallons of caustic solution for each regeneration, or for the series of five regenerations, 125 lbs of NaOH for 465,000 gallons. Assuming a caustic cost of five cents/lb, \$6.25 was required for caustic makeup. $\text{Ca}(\text{OH})_2$ use averaged 28 pounds/regeneration for a total of about \$1.40/465,000 gal. Alumina makeup requirements were very low. In fact, the alumina makeup requirements were so low as to be difficult to measure. No losses were evident upon measuring alumina column heights after approximately five regenerations for each column. The value of 0.2 cent/1000 gal was arbitrarily chosen as an alumina makeup cost, as shown in Table 2. The estimated process operating costs for the pilot plant and laboratory study are presented in Table 2. Though the individual costs have changed, the total cost is about the same as that estimated following the laboratory study. The causes for the increased NaOH makeup requirements and lower alumina losses are unknown at this time.

Differently synthesized aluminas were used in the pilot plant and lab studies, but the effects of this change cannot be realistically evaluated due to differences in hardware size between laboratory and pilot plant and insufficient pilot plant operating time. There is a good possibility of lowering NaOH makeup costs below present values through modifications in operational technique. The first 20 to 40 gal of backwash following elution contains up to 0.7N NaOH. Saving this, rather than diverting it into the 800 gal of solution to be used in column preconditioning preceeding elution, could save about 15% of the present 25 lb NaOH make-up requirement, or about 3.75 lbs of NaOH.

In addition, step (1) of the regeneration cycle could probably be eliminated, and the volume of washwater in the second half of step (5) reduced.

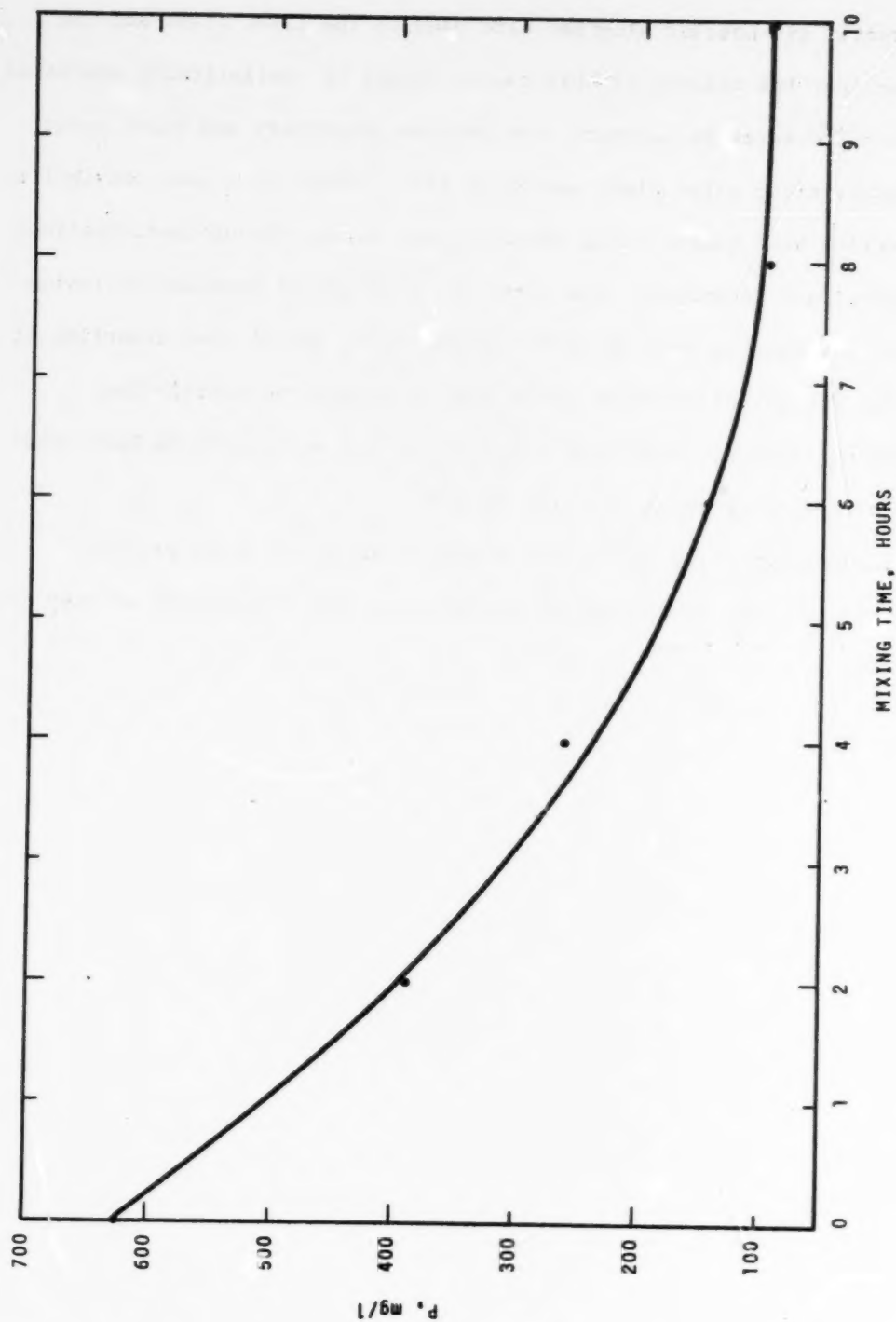


FIGURE 14. Removal of phosphorus vs. time for 800 gal of used eluting solution with 20 lbs of added Ca(OH)_2 .

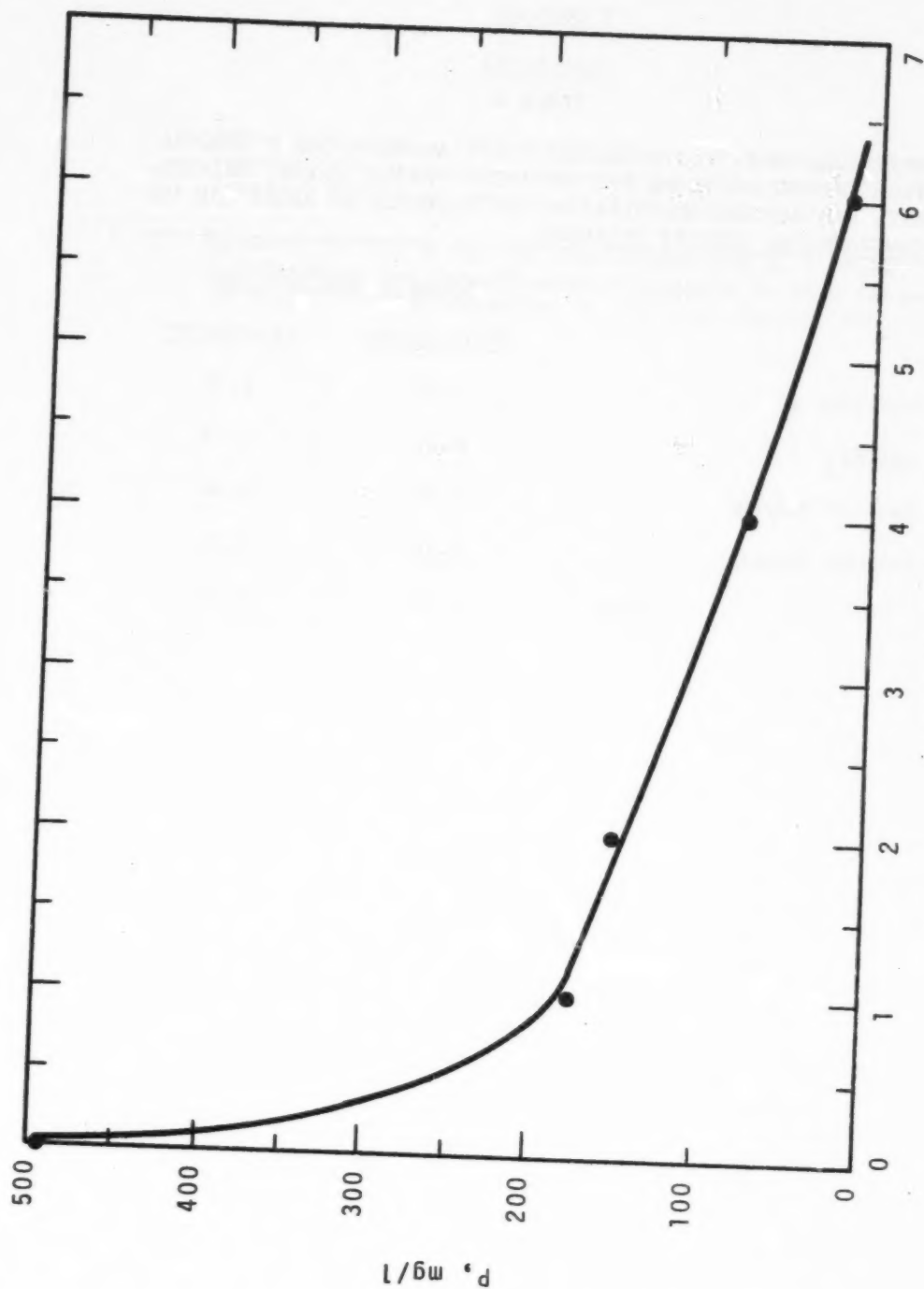


FIGURE 15. Removal of phosphorus vs. time for 800 gal of used eluting solution with 30 lbs of added $\text{Ca}(\text{OH})_2$.

TABLE 2

ESTIMATED MATERIALS OPERATING COSTS ASSUMING 98% P REMOVAL FROM AN AVERAGE 10 mg P/l INFLUENT. POWER, LABOR, MAINTENANCE AND CAPITAL AMORTIZATION COSTS SHOULD BE ADDED FOR THE GEOGRAPHICAL AREA OF INTEREST.

	<u>Costs, cents/1000 gal</u>	
	<u>Pilot Plant</u>	<u>Laboratory</u>
Filtration	1.50	1.50
Ca(OH) ₂	0.31	0.33
Caustic Makeup	1.34	0.40
Alumina Makeup	<u>0.20</u>	<u>1.20</u>
TOTAL	3.35	3.33

SECTION V

REFERENCES

1. Ames, L. L., Jr. "The Use of Alumina Columns for Phosphorus Removal from Secondary Sewage Effluents". Robert A. Taft Research Center Report No. TWRC-8, March, 1969.
2. Harwood, J. E.; R. A. van Steenderen; and A. L. Kühn. "A Rapid Method for Orthophosphate Analysis at High Concentrations in Water." Water Research, 3, 417-423, March, 1969.

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